

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE
Before the Board of Patent Appeals and Interferences

In re Patent Application of

FROOM et al

Serial No. 09/752,834

Filed: January 3, 2001

Atty. Dkt. 608-241

C#/M#

Group Art Unit: 1623

Examiner: Oh, T.

Date: November 12, 2002

Title: ESTER SYNTHESIS

Assistant Commissioner for Patents
Washington, DC 20231

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Sir:

☐ **NOTICE OF APPEAL**

Applicant hereby appeals to the Board of Appeals from the decision dated _____ of the Examiner twice/finally rejecting claims _____ (\$ 320.00)

\$ 0.00

☒ An appeal **BRIEF** is attached in triplicate in the pending appeal of the above-identified application (\$ 320.00)

\$ 320.00

☐ An **ORAL HEARING** is requested under Rule 194 (\$ 280.00) (due within two months after Examiner's Answer)

\$ 0.00

☐ Credit for fees paid in prior appeal without decision on merits

-\$ (0.00)

☐ A reply brief is attached in triplicate under Rule 193(b)

(n fee)

☐ Petition is hereby made to extend the current due date so as to cover the filing date of this paper and attachment(s) (\$110.00/1 month; \$400.00/2 months; \$920.00/3 months; \$1440.00/4 months)

\$ 0.00

SUBTOTAL \$ 310.00

☐ Applicant claims "Small entity" status; enter 1/2 of subtotal and subtract

-\$ (0.00)

☐ "Small entity" statement attached.

SUBTOTAL \$ 310.00

Less month extension previously paid on

-\$ (0.00)

TOTAL FEE ENCLOSED \$ 310.00

Any future submission requiring an extension of time is hereby stated to include a petition for such time extension. The Commissioner is hereby authorized to charge any deficiency, or credit overpayment, in the fee(s) filed, or asserted to be filed, or which should have been filed herewith (or with any paper hereafter filed in this application by this firm) to our **Account No. 14-1140**. A duplicate copy of this sheet is attached.

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NIXON & VANDERHYE P.C.

By Atty.: Leonard C. Mitchard, Reg. No. 29,009

Signature: _____



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APPEAL BRIEF

Sir:

Applicant hereby appeals the Final Rejection of March 27, 2002, Paper No. 13.

REAL PARTY IN INTEREST

The real party in interest is BP Chemicals Limited, a corporation of the United Kingdom.

RELATED APPEALS AND INTERFERENCES

The appellant, the undersigned, and the assignee are not aware of any related appeals or interferences which will directly affect or be directly affected by or have a bearing on the Board's decision in this appeal, except the appeal in commonly assigned copending application Serial No. 09/752,835, filed January 3, 2001.

STATUS OF CLAIMS

Claims 1-15 are pending and have been rejected. No claims have been substantively allowed.

STATUS OF AMENDMENTS

One amendment has been filed since the date of the Final Rejection, namely on September 11, 2002.

SUMMARY OF INVENTION

The invention of the claims relates to a process for producing lower aliphatic esters by reacting an olefin with a lower carboxylic acid in the presence of an acidic catalyst (page 1, lines 1 and 2). More specifically, the process comprises reacting a lower olefin with a saturated lower aliphatic mono-carboxylic acid in the vapor phase in the presence of a heteropoly acid catalyst. The reaction is carried out in a plurality of reactors arranged in series and the feedstock has no more than 0.1 ppm of metallic or metal compound impurities prior to being brought into contact with the heteropoly acid catalyst (page 1 last line through page 2, line 6).

The present invention centers on the discovery that metallic or metal compound impurities present in reactants and any inert gases used in the synthesis of esters by reacting olefins with lower aliphatic carboxylic acids have a tendency to deactivate the acid catalyst. In particular, it has been discovered by the present inventors that the presence of, for example, iron, chromium, molybdenum, and nickel arising from the corrosion of equipment and sodium/potassium (if present in significant amounts) or calcium from any water or acetic acid reactant used are detrimental to the heteropoly acid catalyst. These impurities may contaminate the catalyst either by entrainment in the vapor streams or as gas phase acetate salts in the vapor stream (page 1, third complete paragraph).

ISSUES

The sole issue in this appeal is as follows: whether the claimed invention is unpatentable over Atkins et al (EP 0757027 A1) in view of Nishino et al (Toku-Kai-Hei 7-71907) and Sato (U.S. Patent 4,465,852).

GROUPING OF CLAIMS

Claims 1-15 stand or fall depending on the outcome of the decision of the Board of Appeals and Interferences.

ARGUMENT

Claims 1-15 stand rejected under 35 USC 103(a) as allegedly unpatentable over Atkins et al (EP 0757027) in view of Nishino et al and Sato (U.S. 4,465,852). Reversal of this rejection is respectfully requested.

The process of the present invention is for the production of lower aliphatic esters (e.g. ethyl acetate). The process comprises reacting a lower olefin (e.g. ethylene) with a saturated lower aliphatic mono-carboxylic acid (e.g. acetic acid) in the vapor phase in the presence of a heteropolyacid catalyst. The reaction is carried out in a plurality of reactors arranged in series, and the feedstock is rendered substantially free of metallic or metal compound impurities prior to being brought into contact with the heteropolyacid catalyst.

An important feature of the invention is the discovery that removal of metallic impurities from the feedstock provides a surprising improvement in the catalyst life and in ester productivity. Such impurities can arise from a variety of sources in commercial feedstocks, e.g., from the presence of metal cations in water supplied from local sources, from corrosion of steel pipe-work and reactor vessels around which

components of the feedstock materials are frequently recycled, and from impurities which are frequently present in commercial grade starting materials.

The prior art cited by the Examiner clearly would not lead one of ordinary skill in the art to take any special precautions in relation to the removal of free metal or metal cations in the feedstock to the acid/olefin reaction. While it may be true that one skilled in the art might try to employ starting materials that are reasonably pure, it does not make economic sense for the skilled person to go out of his or her way to use specially purified materials unless there are good reasons to do so. For example, it would be natural for one skilled in the art to use local water (which invariably contains metal ions) for an industrial process, unless specifically instructed to use de-ionised water.

Claim 1 requires that the feedstock be rendered substantially free of metallic impurities prior to contact with the catalyst. Atkins discloses that the catalyst support preferably has less than 0.3% (3000 ppm) by weight of impurities, but Atkins is completely silent as regards the feedstock purity. Claim 1 is clearly patentably distinguished from Atkins et al on this basis alone.

Atkins relates to the same technology as the presently claimed process and therefore represents a good basis for one skilled in the art to consult if wishing to improve a process. Atkins discloses that it is desirable to employ a silica support (for the heteropolyacid catalyst) that is "suitably free from extraneous metals or elements which might adversely affect the catalytic activity of the system". Atkins does not, however, cast any light on what is meant by "extraneous metals or elements". Indeed, in a similar manner to the present specification, Atkins mentions metals that can be present as "counter-ions" (i.e., cations) in the heteropolyacid catalysts (see Atkins Table

at the foot of Page 2 bridging page 3, and associated text), wherein are mentioned sodium, potassium, and cesium salts of the heteropolyacids.

The Examiner seems to be taking the view that the disclosure Atkins concerning the level of impurities in the silica support for the catalyst should extend to industrial processes wherein the catalyst is employed as a component to catalyze industrial organic chemical reactions. However, one skilled in the art would not assume that these two factors are related. The catalyst preparation and its use to promote organic chemical reactions are separate and different chemical processes. They can even be performed, if desired, by different organizations at separate locations. Atkins confines the disclosure of the impurity level to the silica support itself, and does not appear to include even, for example, the heteropolyacids or other materials involved in the catalyst preparation. Thus, one skilled in the art would view this disclosure as the need to avoid the presence of the defined impurities in the support *per se*. There is no suggestion in Atkins that this disclosure should also apply to the feedstocks employed in synthetic organic chemical processes wherein the catalyst is used to promote the desired chemical reaction.

Atkins discloses to the skilled person that the Atkins catalyst should be desirably supported on a silica support prepared using a silica that is free from "extraneous metals or elements". However, there is no suggestion that the feedstock to the reaction need be especially freed from any impurities, and nothing in Atkins would lead the skilled person to conclude that freeing the feedstock from metals would result in special advantages. Accordingly, Atkins falls far short of leading toward the present invention.

Sato fails to give rise to a *prima facie* case of obviousness when combined with Atkins. The introductory paragraph of Sato describes two prior art methods of making esters by reaction of olefins with carboxylic acids. The first type ({1}- relating to the use of strong acids such as sulphuric) is described as having the serious defect of causing corrosion of the apparatus. The second type ({2} - which relates to the use of heteropolyacid catalysts) is said to have the disadvantage of producing (by-product) alcohols and, also, of giving the wrong product when ethylene is employed as the olefin. Thus, if Sato says anything in relation to the technology of the present invention, it is saying, "Don't use it!"

Sato goes on to say that in order to remove the above disadvantages, "a method using various ion exchange resins has been proposed". It is believed that it is far from clear what this means at all. There is no disclosure as to what is meant by this "ion exchange" technology, or as to the purpose of employing ion exchange resins. The reader is left questioning whether these resin catalysts incorporate acid catalysts or whatever? Moreover, there is no disclosure as to whether the use of these "various ion exchange resins" ever met with any success.

Sato then goes on to describe a different type of catalyst based on a crystalline metallosilicate type of catalyst (which is entirely different from the heteropolyacid catalyst of the present invention). Sato is clearly irrelevant to the invention as presently claimed.

The question is: whether it would have been obvious to one of ordinary skill, in the light of the cited prior art, to remove impurities from the feedstock prior to contact with the catalyst, in order to improve the performance of the catalyst. Atkins makes no

mention of the purity of the feedstock, still less that it contains impurities which could adversely affect catalytic performance. Consequently, the person of ordinary skill reading Atkins is given no suggestion that removal of impurities from the feedstock would be a good thing to do. As for Sato, it is simply irrelevant. Sato adds nothing to the disclosure of Atkins. All that Sato suggests is that strong acid catalysts (not used in the present invention) cause corrosion, and that ion-exchange resins could replace heteropolyacids as catalysts (again not a feature of the present invention).

Nishino relates to the preparation of ethyl acetate by the reaction of ethylene and acetic acid over a heteropolyacid catalyst. However, Nishino appears to say nothing about the desirability of removing metal impurities from the starting materials (feedstock); nor, indeed, is anything mentioned about metal contaminants or impurities. Accordingly, Nishino adds nothing of relevance to the question of obviousness.

In light of the above, there would have been no motivation for one of ordinary skill to arrive at the present invention based on the cited art, either when taken singly or in combination. Absent any such motivation, it is clear that no *prima facie* case of obviousness has been made out. Reversal of the obviousness rejection is accordingly respectfully requested.

CONCLUSION

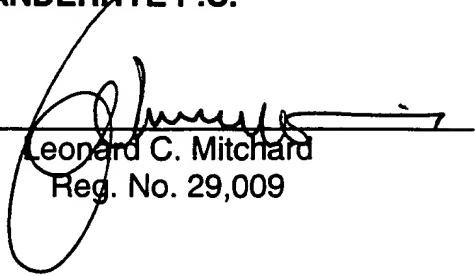
In conclusion it is believed that the application is in clear condition for allowance. Therefore, reversal of the Final Rejection and passage of the subject application to issue are earnestly solicited.

FROOM et al
Serial No. 09/752,834

Respectfully submitted,

NIXON & VANDERHYE P.C.

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APPENDIX

CLAIMS ON APPEAL

1. (Twice amended) A process for the production of lower aliphatic esters which comprises reacting a lower olefin with a saturated lower aliphatic mono-carboxylic acid in the vapour phase in the presence of a heteropolyacid catalyst, wherein a) the reaction is carried out in a plurality of reactors set up in series, and b) the feedstock has no more than 0.1 ppm of metallic or metal compound impurities prior to being brought into contact with the heteropolyacid catalyst.

2 A process as claimed in claim 1, wherein the feedstock has no more than 0.1 ppm of metals and/or metal compounds, prior to being brought into contact with the heteropolyacid catalyst.

3. A process as claimed in claim 1, wherein the feedstock has less than 0.01 ppm of metals and/or metal compounds, prior to being brought into contact with the heteropolyacid catalyst.

4. A process as claimed in claim 1, wherein the feedstock is rendered substantially free of metallic or metal compound impurities which arise from the corrosion of equipment, or which result from any water or acetic acid reactant used in the reaction.

5. A process as claimed in claim 4, wherein the metallic or metal compound impurities which arise from the corrosion of equipment comprise: iron, chromium, nickel and/or molybdenum.

6. A process as claimed in claim 4, wherein the metallic or metal compound impurities which result from any water or acetic acid reactant used in the reaction comprise sodium, potassium and/or calcium.

7. A process as claimed in claim 1, wherein the metallic or metal compound impurities are removed from the feed to the reactor using a guard bed and/or a vaporiser.
8. A process as claimed in claim 7, wherein the guard bed is in the form of an ion-exchange resin.
9. A process as claimed in claim 7, wherein the guard bed comprises amorphous aluminosilicates, clays, zeolites, aluminophosphates, silicoaluminophosphates, metalaluminophosphates or supported heteropolyacids.
10. A process as claimed in claim 1, wherein the metallic or metal compound impurities are removed from the feed to the reactor using a vaporiser which employs demister pads and/or a heavy ends take-off at the base of the vaporiser to remove said impurities.
11. A process as claimed in claim 10, wherein fresh acid is introduced into the vaporiser to scrub out the metallic or metal compound impurities.
12. A process as claimed in claim 1, wherein the saturated, lower aliphatic mono-carboxylic acid reactant is a C1-C4 carboxylic acid.
13. A process as claimed in claim 1, wherein said lower olefin is ethylene.
14. A process as claimed in claim 1, wherein the mole ratio of olefin to the lower monocarboxylic acid in the reactant gases fed to the first reactor is in the range of from 1:1 to 18:1.
15. A process as claimed in claim 1, wherein said plurality of reactors set up in series is in the form of one long reactor which has a plurality of catalyst beds set up in series.